UNIVERSITI TEKNOLOGI MARA

PREPARATION AND CHARACTERIZATION OF LITHIUM BASED GLASS CERAMIC CONDUCTING ELECTROLYTES

MAZIIDAH BINTI HAMIDI

Thesis submitted in fulfillment
of the requirements for the degree of
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AUTHOR'S DECLARATION

I declare that the work in this thesis was carried out in accordance with the regulations of Universiti Teknologi MARA. It is original and is the result of my own work, unless otherwise indicated or acknowledged as referenced work. This thesis has not been submitted to any other academic institution or non-academic institution for any other degree or qualification.

I, hereby, acknowledge that I have been supplied with the Academic Rules and Regulations for Post Graduate, Universiti Teknologi MARA, regulating the conduct of my study and research.

Name of Student	:	Maziidah binti Hamidi
Student I.D. No.	:	2009556763
Programme	:	Master of Science (AS780)
Faculty	:	Faculty of Applied Sciences
Thesis Title	:	Preparation and Characterization of Lithium Based Glass Ceramic Conducting Electrolytes
Signature of Student	:	.....
Date	:	June 2014

ABSTRACT

Lithium aluminium titanium phosphate (LATP), $Li_{1+x}Al_xTi_{2-x}(PO_4)_3$ (x = 0.0-0.5), lithium based glass ceramics conducting electrolyte with NASICON were successfully prepared by sol gel technique with acetic acid as chelating agent. TGA was carried out to determine the annealing temperature by finding out the precursors' thermal stability. $Li_{1+x}Al_xTi_{2-x}(PO_4)_3$ were annealed at 600°C, 700°C, 800°C and 900°C for 3 hours in air. XRD revealed major phase of $LiTi_2(PO_4)_3$, which has NASICON structure, exist in all prepared samples with secondary phases TiP_2O_7 and $AlPO_4$. FTIR shows presence of NASICON phosphate peaks from which dominates wavenumber ranged 600 cm^{-1} to 1300 cm^{-1}. Peaks around 1045 cm^{-1}, 870 cm^{-1}, 740 cm^{-1}, and 610 cm^{-1} indicate the presence of NASICON PO_4^{3-} in all of the prepared samples. This also confirms the presence of $LiTi_2(PO_4)_3$ in all of the samples. The ionic conductivity were analyzed with IS at room temperature and elevated temperature. The highest room temperature conductivity was 2.84 x 10^{-5} Scm^{-1} of sample $Li_{1.4}Al_{0.4}Ti_{1.6}(PO_4)_3$, (LATP 4), annealed at 800°C for 3 hours in air. The increase in ionic conductivity from LATP 0 annealed at 800°C, 3.12 x 10^{-6} Scm^{-1} shows that the partial substitution of Al^{3+} with Ti^{4+} was able to increase the ionic conductivity. The high ionic conductivity of LATP 4 was supported by the lower intensity of impurity peaks, as reported in XRD. From ac conductivity analysis using Jonscher's universal power law, the s values of LATP 4 at various temperatures are within the range $0 < s < 1$. It is concluded that the LATP 4 annealed at 800°C followed the quantum mechanical tunneling (QMT) model due to the independency of s to temperature.

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